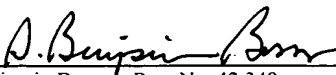# UNITED STATES UTILITY PATENT APPLICATION FOR

SURFACES HAVING PARTICLE STRUCTURES WITH

BROAD RANGE RADIATION ABSORPTIVITY

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SURFACES HAVING PARTICLE STRUCTURES WITH BROAD RANGE RADIATION ABSORPTIVITY

5   **Related Applications**

This application claims priority under 35 U.S.C. §120 to United States Utility Patent Application Serial No: 09/669,796, filed September 26, 2000, which claimed priority under 35 U.S.C. §119 to United States Provisional Patent Application Serial No: 60/156,471, now abandoned. This application is related to United States Utility Patent

10   Application Serial No: 09/670,453, filed September 26, 2000, United States Utility Patent Application Serial No: 09/669,369, filed September 26, 2000 and United States Utility Patent Application titled "Amplification of Analyte Detection by Substrates Having Particle Structures With Receptors", inventors David I. Kreimer, Oleg A. Yevin and Thomas H. Nufert, filed March 23, 2001. All of those Patent Applications are herein

15   incorporated fully by reference.

BACKGROUND OF THE INVENTION

Field of the Invention

This invention relates to the manufacture of surfaces having improved heat

20   radiation absorptivity. Specifically, the invention relates to the manufacture of windows and heat pipes with surfaces involved in absorption and radiation of heat via radiative heat transfer. More specifically, the invention relates to the manufacture of heat pipes, windows and window materials with surfaces covered with nanoparticle structures for absorption of broad band electromagnetic radiation.

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Description of Related Art

Many useful mechanical and electrical processes inside structures result in dissipation of energy in the form of heat. This heat is often undesirable, because it causes increase in the temperature of an structure, such as a building or increases the temperature

30   of sensitive equipment. Additionally, in the use of solar radiation as a source of energy, heating up of solar-radiation absorption elements is the first step in energy conversion.

At the next steps, heat pipes are often used for rapid heat transfer from the solar-radiation absorption elements to other elements in these systems.

In general, a heat pipe transfers heat faster than a solid copper rod of the same diameter. Heat pipes transfer heat with little loss, and do not require the input of additional sources of energy, such as compressors. This capacity to transfer heat without the need of energy input supports broad technological applications for heat pipes. To prevent heating up the structure, windows and window materials have been applied to the window to either permit or hinder transfer of radiation from one side of the window to another.

Additionally, in the use of solar radiation as a source of heating structures, transmission of radiant energy through the window into a structure is a first step. Within the structure, the radiant energy can be absorbed by elements within, and converted into thermal energy as manifested by increased temperature. Some thermal energy can be dissipated as radiation of longer wavelengths, including those in the infrared range. To promote efficient heating, it can be desirable to permit easy passage of incident radiation, but hinder exit of long wavelength radiation. Untreated glass or plastics can exhibit desirable radiation transfer properties, and is extensively used in greenhouses. Glass is widely used for windows. However glass is thermally conductive and can conduct electromagnetic radiation. Because substantial radiative heat can be lost through glass windows by the passage of electromagnetic radiation, it is desirable to provide glass for windows having reduced transmissability to certain wavelengths of electromagnetic radiation.

I.   **Heat Pipes**

Figure 1 illustrates the principle of operation of a typical heat pipe **100**, which is a hollow metal tube **105** sealed at both ends, with a wick **135** covering the inner surface of the tube. The tube is filled with a volatile fluid **140**. When one end of the pipe **110** is heated up, as indicated by Qin, the volatile fluid **140** evaporates at this end of the tube. The vapor expands and moves to the other, cooler end **120** of the tube (curved arrows), where condensation of the volatile fluid occurs, releasing heat out (Qout). The condensate moves back to the hot end of the pipe via the wick path **135** due to capillary

forces. The cycle of evaporation at a warm end at which the heat energy is absorbed from the environment and condensation at a cool end, thereby releasing the heat to the environment, is thus the basis for heat transfer in heat pipes.

Typically, the external tube is made of materials with high thermal conductivity,

5   e.g., copper. These materials provide absorption and/or emission of heat under conditions of contact and convective heat transfer. However, when a radiative heat transfer mechanism is involved, high heat conductivity, by itself, is insufficient for desired highly efficient heat transfer, and other factors can improve the efficiency of the process. These requirements are: 1) the external surface of the pipe should absorb and/or

10   emit electromagnetic radiation in a broad wavelength range; and 2) the transfer of heat between the bulk of the tube material and the external surface should be efficient. One approach to manipulate absorptive and/or emissive characteristics of surfaces is done by their texturing with micron-sized geometric holes and coating the pipes with materials having a desirable emission spectrum, such as tungsten (Stone, et al., U.S. Patent No:

15   5,932,029, herein incorporated fully by reference).

Glass and plastic are widely used for windows and window coverings. However, untreated glass and plastics can conduct electromagnetic radiation. Because substantial radiative heat can be lost through glass windows by the passage of electromagnetic radiation, it is desirable to provide materials for windows having reduced transmissability

20   to certain wavelengths of electromagnetic radiation, while maintaining transmissability to other wavelengths.

SUMMARY OF THE INVENTION

Thus, one object of this invention is the development of surfaces with superior

25   capacity to absorb and emit electromagnetic radiation in a broad spectral range by means of a nanoparticle structure within the material or applied to the surface of the material.

Another object of this invention is the manufacture of surfaces having nanoparticle structures having broad range heat absorptive and/or emissive properties.

Yet another object of this invention is the manufacture of surfaces having

30   nanoparticle structures having specific range heat absorptive and/or emissive proprieties.

These and other objects are met by design and manufacture of a surfaces having nanoparticle structures as part of the structure. In certain embodiments, heat pipes are covered with particle structures that are capable of effective absorption and/or emission of electromagnetic radiation. These heat pipes are herein termed "Radiation Heat Pipes."

5   In certain embodiments of this invention, surfaces are created that are capable of absorption and/or emission of light in desirable spectral ranges via manipulations of the properties of fractal structures by means of photomodification, by varying the size, and distances between the nanoparticles, by varying the shape of particles, the material of particles and/or the material of the thermal conductive layer and/or material of the tube

10   of the heat pipe.

In other embodiments, a sequence of chemical reactions can be performed to form particles having defined spatial relationships with each other, which, in turn, are used to produce particle structures. In a set of chemical reactions, metal particles can be reacted with molecular linkers. These reactions can proceed nearly to completion if the affinities

15   of reactants are sufficiently high and if the ratios of reactants are controlled. The products of these reactions can include various suspensions of particle structures of a characteristic size as pre-determined by the lengths of the linkers.

Another aspect of this invention is the use of ridged linkers of various lengths to maintain a characteristic size of the particle structures. Standard linker lengths allow a

20   degree of control of particle structure formation and manipulation of properties of the particle structures. In a first linking reaction, the use of linker molecules of relatively short fixed lengths allows metal particles to be positioned at fixed distances from one another. In subsequent reactions, the lengths of the linkers can be increased, so as to provide a longer length between pairs of particles. By using the set of products described

25   herein, particle structures can be generated in a controllable fashion, for example in the manufacture of heat pipes having either broad band absorbance and/or emission or, if desired, narrow band absorbance and/or emission.

Another aspect of this invention comprises window materials and window coverings. In these embodiments, particle structures having desired radiation absorptivity

30   characteristics are made to permit improved heating and/or cooling of interior spaces having such windows. In certain embodiments, window materials have particle structures

on a surface, either as permanent or temporary adjuncts to the window material. In certain of these embodiments, particle structures can be sprayed onto the surface of a window material to confer the desired properties of radiation absorptivity to the window. In alternative embodiments, particles structures can be applied in the form of a tape or

5   polymer having particle structures embedded within the polymer. The polymer can be attached to the window material with adhesive, and in certain embodiments can be easily removed. In yet other embodiments, window materials and window coverings can be made with particle structures and other adjuncts, such as dyes or polarizers.

In yet other embodiments, window materials can be made and used with optical

10   devices used for analyte detection. Cuvettes, tubes, slides and the like can be prepared and can have desirable radiation absorptive characteristics, so as to decrease effects of undesired radiation, including parasite radiation.

BRIEF DESCRIPTION OF THE DRAWINGS

15   The invention will be described with respect to the particular embodiments thereof. Other objects, features and advantages of the invention will become apparent with reference to the specification and drawings in which:

Figure 1 is a drawing depicting a design for a heat pipe of the prior art and a mechanism of heat transfer.

20   Figures 2a to 2c depict schematically a set chemical reactions for manufacturing particle structures of this invention.

Figure 3 depicts a detailed design of the radiation absorbing end of a heat pipe of this invention.

Figure 4 depicts a heat pipe of this invention designed for collecting

25   electromagnetic radiation in a broad spectral range, converting this energy into heat and transfer this heat to a cooled end of the pipe.

Figure 5 depicts a design for a heat pipe of this invention capable of both heat absorption and heat radiation via radiative heat transfer.

Figure 6 depicts a design for window glass having particle structures thereon.

30   Figure 7 depicts a graph of optical density of a piece of window glass having fractal aggregates thereon.

Figures 8a - 8b depict alternative embodiments of this invention in which particle structures are within window materials.

Figures 9a - 9b depict embodiments of this invention in which window coverings have particle structures.

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DETAILED DESCRIPTION OF THE INVENTION

Definitions

10   The following words and terms are used herein.

      The term "fractal" as used herein means a structure comprised of elements, and having a relationship between the scale of observation and the number of elements, i.e., scale-invariant. By way of illustration only, a continuous line is a 1-dimensional object. A plane is a two-dimensional object and a volume is a three-dimensional object.

15   However, if a line has gaps therein, and is not a continuous line, the dimension is less than one. For example, if ½ of the line is missing, then the fractal dimension is ½. Similarly, if points on a plane are missing, the fractal dimension of the plane is between one and 2. If ½ of the points on the plane are missing, the fractal dimension is 1.5. Moreover, if ½ of the points of a solid are missing, the fractal dimension is 2.5. In scale

20   invariant structures, the structure of objects appears to be similar, regardless of the size of the area observed. Thus, fractal structures are a type of ordered structures, as distinguished from random structures, which are not ordered.

      The terms "fractal aggregate" or "fractal associate" as used herein, means a structure of limited size, comprising at least about 100 individual particles associated

25   together, and which demonstrates scale invariance within an area of observation limited on the lower bound by the size of the individual particles comprising the fractal associate and on the upper bound by the size of the fractal associate.

      The term "fractal dimension" as used herein, means the exponent D of the following equation: $N \propto R^D$, where R is the area of observation, N is the number of

30   particles, and D is the fractal dimension. Thus in a non-fractal solid, if the radius of observation increases by 2-fold, the number of particles observed within the volume

increases by 2^3. However, in a corresponding fractal, if the radius of observation increases by 2-fold, the number of particles observed increases by less than 2^3.

The term "fractal particle associates" as used herein means a large number of particles arranged so that the number of particles per unit volume (the dependent variable) or per surface unit changes non-linearly with the scale of observation (the independent variable).

The term "linker" as used herein means an atom, molecule, moiety or molecular complex having two or more chemical groups capable of binding to a surface and permitting the attachment of particles together to form groups of particles. The simplest linker connects two particles. A branched linker may link together larger numbers of particles.

The term "ordered structures" as used herein means structures that are non-random.

The term "particle structures" as used herein means a group of individual particles that are associated with each other in such a fashion as to permit enhancement of electric fields in response to incident electromagnetic radiation. Examples of particles include metals, metal-coated polymers and fullerenes. Also included in the meaning of the term "particle structures" are films or composites comprising particles on a dielectric surface or imbedded in a dielectric material.

The term "percolation point" as used herein means a point in time on a conductive surface or medium when the surface exhibits an increase in conductance, as measured either via surface or bulk conductance in the medium. One way to measure surface or "sheet" conductance is via electric probes applied to the surface.

The term "random structures" as used herein means structures that are neither ordered nor fractal. Random structures appear uniform regardless of the point and scale of observation, wherein the scale of observation encompasses at least a few particles.

The term "resonance" as used herein means an interaction with either incident, scattered and/or emitted electromagnetic radiation and a surface having electrons that can be excited by the electromagnetic radiation and increase the strength of the electric field of the electromagnetic radiation.

The term "resonance domain" as used herein means an area within or in proximity to a particle structure in which an increase in the electric field of incident electromagnetic radiation occurs.

The term "scaling diameter" as used herein means a relationship between particles in a nested structure, wherein there is a ratio (scaling ratio) of particle diameters that is the same, regardless of the size of the particles.

Embodiments of the Invention

This invention includes the use of particle structures on the absorbent and/or emissive portions of heat pipes. The nanoparticle structures can be applied to the exterior surfaces of a heat pipe and can thereby efficiently absorb or emit heat energy. By increasing the efficiency of absorbance by the exterior surface of the heat pipe, heat energy can be more easily transmitted to the interior of the heat pipe, and can therefore increase the rate of evaporation of the volatile fluid in the interior of the heat pipe. The increased evaporation of the volatile fluid can result in increased flow of evaporated fluid, thereby permitting increased heat flow from the absorptive portion of the heat pipe to the emissive portion of the heat pipe. The increased flow of heat to the emissive portion can result in increased heat loss from that portion of the pipe. In other embodiments of the invention, the emissive portion of the heat pipe can also be coated with a particle structure to permit the increased loss of heat from the emissive portion of the heat pipe.

Window materials, including glass, plastic and other materials known in the art can have improved heat retention or transmission and can be made by applying nanoparticle structures to a surface of the material or can be incorporated within the matrix of the the material. In other embodiments, a multilayered or "sandwich" structure can be made in which a first layer of window material can be provided, a layer of material having particle structures is provided thereon and an additional layer of window material can be applied. For window materials used in homes and/or other structures through which it is desirable to be able to see, the particle structures can desirably by provided that have relatively low absorptivity in the visible wavelength range, yet have greater absorptivity at wavelengths outside the visible range.

In certain embodiments, nanoparticle structures can be temporarily applied to the widow materials' surface. Such use can be desirable in climates in which cold weather promotes radiative heat loss through windows of a structure, for example, a building. When the outside temperature is low, one can apply a coating of particle structures to the window to absorb and therefore retain radiant energy arising from within the structure having the window. When the temperature outside the structure increases, the coating can be removed if desired to permit escape of excess heat via radiant mechanisms. Because radiant energy generated inside a structure characteristically has long wavelengths, it can be desirable to coat a window with nanoparticle structures having high absorption at longer wavelengths, for example having wavelengths in the infrared to visible range.

Alternatively, in warm climates, it can be desirable to decrease the amount of radiation entering through a window in a structure. In these situations, one can apply a coating having nanoparticle structures that absorb the incoming radiant energy. Because the radiant energy does not penetrate into the structure, the amount of heat entering the structure can be decreased. When the outside temperature is reduced and it is desirable to increase the amount of incoming radiation, one can remove the coating and thereby permit radiation to enter the structure.

In addition to providing windows for structures, this invention also includes coatings for smaller structures, for example tubes, slides, and other devices in which optical absorbance and/or transmission properties are desired. For example, in certain embodiments of this invention, an optical cuvette or sample holder can be provided having decreased transmittance for certain wavelengths that might interfere with detection of electromagnetic radiation, or "parasite radiation" as described in co-pending U.S. Application Serial No: 09/669,369, filed September 26, 2000, incorporated herein fully by reference. Such embodiments can be also desired for detection of analytes as described in co-pending U.S. Patent Application Serial No: 09/670,453, filed September 26, 2000, incorporated herein fully by reference.

The structures that are desirable for use according to the methods of this invention include structures of small particles in structures, herein termed particle structures, which includes as a subset, fractal associates. Particle structures can be characterized by having

physical and chemical structures that enable oscillations of electrons to be in resonance with incident and outgoing electromagnetic radiation.

I. Manufacture of Particle Structures

5   The particle structures desirable for use according to this invention can include any structure in which electromagnetic signals can be absorbed or emitted across a wide range of wavelengths. The following discussion regarding metal particle structures is not intended to be limiting to the scope of the invention, but is for purposes of illustration only. Other structures, including fractal structures can be desirable.

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A. Manufacture of Metal Particles

To make metal particles for according to some embodiments of this invention, we can generally use methods known in the art. Tarcha et al., U.S. Patent No: 5,567,628, incorporated herein fully by reference. Metal colloids can be composed of noble metals,

15   specifically, elemental gold or silver, copper, platinum, palladium and other metals known to provide broad band absorption and emission in a desirable spectral range. In general, to make a metal colloid, a dilute solution containing the metal salt is chemically reacted with a reducing agent. Reducing agents can include ascorbate, citrate, borohydride, hydrogen gas, and the like. Chemical reduction of the metal salt can

20   produce elemental metal in solution, which combine to form a colloidal solution containing metal particles that are relatively spherical in shape.

Example 1
Manufacture of Gold Colloid and Fractal Structures

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In one embodiment of this invention, a solution of gold nuclei is made by preparing a 0.01% solution of $NaAuCl_4$ in water under vigorous stirring. One milliliter ("ml") of a solution of 1% sodium citrate is added. After 1 minute of mixing, 1 ml of a solution containing 0.075 % $NaBH_4$ and 1% sodium citrate is added under vigorous

30   stirring. The reaction is permitted to proceed for 5 minutes to prepare the gold nuclei having an average diameter of about 2 nm). The solution containing the gold nuclei can be refrigerated at 4° C until needed. This solution can be used as is, or can be used to

produce particles of larger size (e.g., up to about 50 nm diameter), by rapidly adding 30 μl of the solution containing gold nuclei and 0.4 ml of a 1% sodium citrate solution to the solution of 1% $HAuCl_4 \cdot 3H_2O$ diluted in 100 ml H_2O, under vigorous stirring. The mixture is boiled for 15 minutes and is then cooled to room temperature. During cooling,

5   the particles in the solution can form fractal structures. The resulting colloid and/or fractal particle structures can be stored in a dark bottle.

Deposition of enhancing particles on dielectric surfaces including glass can generate films that can enhance electromagnetic signals. Such films can be as thin as about 10 nm. In particular, the distribution of electric field enhancement on the surface

10   of such a film can be uneven. Such enhancing areas are resonance domains. Such areas can be particular useful for positioning receptors for analyte binding and detection. For films or particle structures embedded in dielectric materials, one way to manufacture enhancing structures is to treat the surface until "percolation points" appear. Methods for measuring sheet resistance and bulk resistance are well known in the art.

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Example 2
Manufacture of Metal Particles and Fractal Structures Using Laser Ablation

In addition to liquid phase synthesis described above, laser ablation is used to

20   make metal particles. A piece of metal foil is placed in a chamber containing a low concentration of a noble gas such as helium, neon, argon, xenon, or krypton. Exposure to the foil to laser light or other heat source causes evaporation of the metal atoms, which, in suspension in the chamber, can spontaneously aggregate to form fractal or other particle structures as a result of random diffusion. These methods are well known in the

25   art.

B.   Manufacture of Films Containing Particles

To manufacture substrates containing metal colloidal particles of one embodiment of this invention, the colloidal metal particles can be deposited onto quartz slides as

30   described in Examples 1 or 2. Other films can be made that incorporate random structures, non-fractal ordered structures and/or fractal aggregates in similar fashions.

Example 3
Manufacture of Quartz Slides Containing Gold Fractal Structures

Quartz slides (2.5 cm x 0.8 cm x 0.1 cm) are cleaned in a mixture of $HCl:HNO_3$

5 (3:1) for several hours. The slides are then rinsed with deionized H_2O (Millipore

Corporation) to a resistance of about 18 $M\Omega$ and then with CH_3OH. Slides are then

immersed for 18 hours in a solution of aminopropyltrimethoxysilane diluted 1:5 in

CH_3OH. The slides are then rinsed extensively with CH_3OH (spectrophotometric grade)

and deionized H_2O prior to immersion into colloidal gold solution described above. The

10 slides are then immersed in the gold colloid solution above. During this time, the gold

colloid particles can deposit and can become attached to the surface of the quartz slide.

After 24 hours, colloid derivatization is complete. Once attached, the binding of colloidal

gold nanocomposites to the quartz surfaces is strong and is essentially irreversible.

During the procedure, ultraviolet and/or visual light absorbance spectra of such

15 derivatized slides are used to assess the quality and reproducibility of the derivatization

procedure. The manufacturing process is monitored using electron microscopy to assess

the density of the colloidal coating, the distribution of gold colloid particles on the

surface, and the size of the gold colloid particles.

20

C. Aggregation of Particles to Form Particle Structures

According to other embodiments of this invention, several methods can be used

to form particle structures. It is known that metal colloids can be deposited onto surfaces,

25 and when aggregated can form fractal structures having a fractal dimension of about 1.8.

Safonov et al., *Spectral Dependence of Selective Photomodification in Fractal

Aggregates of Colloidal Particles,* Physical Review Letters 80(5):1102-1105 (1998)

incorporated herein fully by reference. Figure 1 depicts a particle structure suitable for

use with the methods of this invention. The particles are arranged in a scale-invariant

30 fashion, which promotes the formation of resonance domains upon illumination by laser

light.

In addition to fractal structures, ordered non-fractal structures and random structures can be generated. These different types of structures can have desirable properties for enhancing signals associated with detection of analytes using electromagnetic radiation.

5   To make ordered non-fractal structures, one can use, for example, chemical linkers having different lengths sequentially as described in more detail below. In addition, using linkers of the same size, one can generate ordered structures, which can be useful for certain applications.

In certain embodiments of this invention, particles can be attached together to

10   form structures having resonance properties. In general, it can be desirable to have the particles being spheres, ellipsoids, or rods. For ellipsoidal particles, it can be desirable for the particles to have a long axis (x), another axis (y) and a third axis (z). In general, it can be desirable to have x be from about 0.05 to about 1 times the wavelength (λ) of the incident electromagnetic radiation to be used. For rods, it can be desirable for x to

15   be less than about 4 λ, alternatively, less than about 3 λ, alternatively less than about 2 λ, in other embodiments, less than about 1λ, and in yet other embodiments, less than about ½ λ. The ends of the rods can be either flat, tapered, oblong, or have other shape that can promote resonance.

For two particle structures, it can be desirable for the particle pair to have an x

20   dimension to be less than about 4 λ, alternatively, less than about 3 λ, alternatively less than about 2 λ, in other embodiments, less than about 1λ, and in yet other embodiments, less than about ½ λ.

For two-dimensional structures, pairs of particles, rods, rods plus particles together can be used. The arrangement of these elements can be randomly distributed,

25   or can have a distribution density that is dependent upon the scale of observation in a non-linear fashion.

In other embodiments, rods can be linked together end-to end to form long structures that can provide enhanced resonance properties.

For three-dimensional structures, one can use regular nested particles, or chemical

30   arrays of particles, associated either by chemical linkers in a fractal structure or in ordered, nested arrays.

In yet other embodiments, of third-order structures, a suspension of particles can be desirable. In certain of these embodiments, the suspended particles can have dimensions in the range of about ½ λ to about 1 millimeter (mm).

Using the strategies of this invention, a researcher or developer can satisfy many needs, including, but not limited to selecting the absorbance of electromagnetic radiation by particle elements, the nature of the surface selected, the number of resonance domains, the resonance properties, the wavelengths of electromagnetic radiation showing resonance enhancement, the porosity of the particle structures, and the overall structure of the particle structures, including, but not limited to the fractal dimensions of the structure(s).

1. Photoaggregation

Photoaggregation can be used to generate particle structures that have properties which can be desirable for use in broad band electromagnetic radiation absorbers.

Irradiation of fractal metal nanocomposites by a laser pulse with an energy above a certain threshold leads to selective photomodification, a process that can result in the formation of "dichroic holes" in the absorption spectrum near the laser wavelength (Safonov et al., Physical Review Letters 80(5):1102-1105 (1998), incorporated herein fully by reference). Selective photomodification of the geometrical structure can be observed for both silver and gold colloids, polymers doped with metal aggregates, and films produced by laser evaporation of metal targets.

One theory for the formation of selective photomodification is that the localization of optical excitations in fractal structures are prevalent in random nanocomposites. According to this theory, the localization of selective photomodification in fractals can arise because of the scale-invariant distribution of highly polarizable particles (monomers). As a result, small groups of particles having different local configurations can interact with the incident light independently of one another, and can resonate at different frequencies, generating different domains, called herein "optical modes." According to the same theory, optical modes formed by the interactions between monomers in fractal are localized in domains that can be smaller than the optical wavelength of the incident light and smaller than the size of the clusters

of particles in the colloid. The frequencies of the optical modes can span a spectral range broader than the absorption bandwidth of the monomers associated with plasmon resonance at the surface. However, other theories may account for the effects of photomodification of fractal structures, and this invention is not limited to any particular theory for operability.

Photomodification of silver fractal aggregates can occur within domains as small as about 24 x 24 x 48 nm^3 (Safonov et al., Physical Review Letters 80(5):1102-1105 (1998), incorporated herein fully by reference). The energy absorbed by the fractal medium can be localized in a progressively smaller number of monomers as the laser wavelength is increased. As the energy absorbed into the resonant domains increases, the temperature at those locations can increase. At a power of 11 mJ/cm^2, light having a wavelength of 550 nm can produce a temperature of about 600 K (Safonov et al., Physical Review Letters 80(5):1102-1105 (1998), incorporated herein fully by reference). At this temperature, which is about one-half the melting temperature of silver, sintering of the colloids can occur (Safonov et al., Id.) incorporated herein fully by reference), thereby forming stable fractal nanocomposites.

As used in this invention, photoaggregation can be accomplished by exposing a metal colloid on a surface to pulses of incident light having a wavelengths in the range of about 400 nm to about 2000 nm. In alternative embodiments, the wavelength can be in the range of about 450 nm to about 1079 nm. The intensity of the incident light can be in the range of about 5 mJ/cm^2 to about 20 mJ/cm^2. In an alternative embodiment, the incident light can have a wavelength of 1079 nm at an intensity of 11 mJ/cm^2.

Fractal aggregates that are especially useful for the present invention can be made from metal particles having dimensions in the range of about 10 nm to about 100 nm in diameter, and in alternative embodiments, about 50 nm in diameter. A typical fractal structure of this invention is composed of up to about 1000 particles.

Figure 2 depicts a particle structure that have been photoaggregated and that are suitable for use with the methods of this invention. Local areas of fusion of the metal particles can be observed (circles).

II.   Particle Structures

In certain embodiments of this invention, the particle structures of this invention can have certain properties of fractals. Fractals are structures which display a pattern of self-similarity. Self-similarity means that their overall structure is scale-invariant in that the structure appears similar over a wide range of magnifications. Fractal-like structures

5   are widely present in nature, by way of example only, clouds. Fractal objects can be also generated artificially. For example, when the landscape of metal surfaces is arranged in the shape of self-similar triangles or other shapes, such fractal objects can serve as a so called "fractal antenna." Such antennas can allow a broader range of radio waves reception and transmission than antennas having more regular structures.

10   Analogously, when metal particles having diameters of about 1/10 the wavelength of the incident light are arranged on a surface in the form of a fractal-like structure, such fractal surface can display absorbance in a broad optical range (Shalaev V. M., et.al. J. Nonlinear Optical Physics & Materials 7(1):131-152 (1998), incorporated herein fully by reference). In this case, fractal-like structure can be due to the fact that the number of

15   particles per a surface area decreases upon increase in the scale of such area. One theory to account for the absorbance of such fractal surfaces is the interference of oscillating dipole moments induced by the electromagnetic waves in individual particles. According to one theory, incident photons induce a field across a particle and thereby can cause mobile electrons in the metal to move with a frequency of the incident electromagnetic

20   field. Such collective movement is referred to herein as a "plasmon wave." According to one theory, the collective oscillation of electrons occurs due to strong dipolar and multipolar interactions of plasmon waves in metal particles within a fractal structure. Because almost any distances and orientations of metal particles can be present in a such fractal system, a large number of possible "resonant cavities" are formed within such

25   structures. By the term "resonant cavity," we mean an arrangement of particles which provides resonance conditions for the wavelengths of incident light. Each of such cavities absorbs and/or emits electromagnetic wave at a specific set of wavelengths,. Because many different resonance cavities can be present in a nanoparticle structure, many different frequencies can be absorbed and/or emitted by the structure. Thus, the

30   collective capacity of all such cavities to resonate within a broad range of wavelengths yields a broad absorption band of such fractal structures.

Chemical reduction of metal colloidal solutions, laser ablation of surfaces, surface etching, annealing of films, are, by way of example only, used for generation of fractal-like structures (U.S. Patent Application Serial No: 09/670,453, filed September 26, 2000, U.S. Patent Application Serial No: 09/669,369, filed September 26, 2000,

5   United States Utility Patent Application titled "Amplification of Analyte Detection by Substrates Having Particle Structures With Receptors", inventors David I. Kreimer, Oleg A. Yevin and Thomas H. Nufert, filed March 23, 2001; and Shalaev et. al. (1998), all references incorporated herein fully by reference). Typically fractal-like structures can be obtained by allowing a far-from-equilibrium system to attain a low energy state. In

10   such systems fractal-like structures can form spontaneously.

In addition to fractal structures, other types of particle structures can result in the enhanced absorptive and/or emissive properties of this invention.

The manufacture of particle structures that can be applied to the surface of the heat pipe or window material is described herein below.

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A.   Chemically Directed Synthesis of Particle Structures

In certain embodiments of this invention, nanoparticle structures can be made using chemical methods. First, metal particles can be either made according to methods described above, or alternatively can be purchased from commercial suppliers

20   (NanoGram Inc., Fremont, California). Second, the particles can be joined together to form first-order structures, for example, pairs of particles. Then, the first-order structures can be joined together to form second-order structures, for example, pairs of particle pairs. Finally, third-order fractal structures can be made by joining second-order structures together.

25   In alternative embodiments of this invention, the formation of a fractal array of metal particles can be carried out using chemical methods. Once metal colloid particles have been manufactured, each particle can be attached to a linker molecule via a thiol or other type of suitable chemical bond. The linker molecules then can be attached to one another to link adjacent colloid particles together. The distance between the particles is

30   a function of the total lengths of the linker molecules. It can be desired to select a stoichiometric ratio of particles to linker molecules. If too few linker molecules are used,

then the array of particles will be too loose or may not form at all. Conversely, if the ratio of linker molecules to particles is too high, the array may become too tight, and may even tend to form crystalline structures, which are not random, and therefore will not tend to promote Raman resonance.

In general, it can be desirable to perform the linking procedure sequentially, wherein the first step comprises adding linker molecules to individual particles under conditions that do not permit cross-linking of particles together. By way of example only, such a linker can comprise an oligonucleotide having a reactive group at one end only. During this first step, the reactive end of the oligonucleotide can bind with a metal particle, thereby forming a first particle-linker species, and having a free end of the linker. The ratio of linker molecules to particles can be selected, depending on the number of linker molecules are to be attached to the particle. A second linker can be attached to another group of particles in a different reaction chamber, thereby resulting in a second linker-particle species, again with the linker having a free end.

After those reactions have progressed, the different linker-particle species can be mixed together and the linkers can attach together to form "particle pairs" joined by the linker molecules. By way of example, Figure 2a to 2c illustrates methods for manufacturing fractal structures of this invention. In Figure 2a, metal particles 10 are formed using methods previously described. Short linkers 20 have chemically active ends that are capable of binding to metal particles 10. For example, linker 20 has sulfhydryl ("SH") groups at each end of the linker 20. When combined, metal particles 10 bind with the SH ends of linkers 20 to form particle pairs 30.

Figure 2b illustrates the steps that can be used to form clusters of particle pairs. Particle pairs 30 are reacted with medium-length linkers 40 to form clusters 50.

Figure 2c illustrates the steps that can be used to form nanoscale fractal structures of this invention. Clusters 50 are reacted with long linkers 60 to form nanoscale fractal structure 70.

Linker molecules can be selected to provide any desired length. Typically, polymers of organic moieties can be useful. For example, linking can be carried out using an aryl di-thiol or di-isonitrile molecules. Alternatively one can use any active moiety that can be used to attach the linker to the metal particle. It can be desirable to

use the above types of aryl linkers with nucleic acid or other types of linker molecules. The linker can have a central area having ethylbenzene moieties, where n is a number between 1 and about 10,000.

In general, the ratio of length for each subsequent pairs of linkers can be in the range of about 2 to about 20. Alternatively, the ratios of lengths of subsequent pairs of linkers can be in the range of about 3 to about 10, and in other embodiments, about 5.

For example, for a three-order manufacturing process involving linkers 1, 2, and 3 (L1, L2, and L3, respectively), it can be desirable for the ration of L1:L2:L3 to be in the range of about 1:2:4. Alternatively, the ratio can be about 1:5:25, and in yet other embodiments, the ratio can be about 1:20:400. In other embodiments, the ratio between L1 and L2 and from L2 to L3 need not be the same. Thus, in certain embodiments the ration of L1:L2:L3 can be 1:3:20, or alternatively, 1:20:40.

Under these conditions, one can manufacture structures having any desired porosity. In general, the size of the nanoscale structures should have average dimensions in the range of about 20 nm to about 10,000 nm. In alternative embodiments, the dimensions can be in the range of about 50 nm to about 300 nm, and in other embodiments, in the range of about 100 to about 200 nm, and in yet other embodiments, about 150 nm.

For certain applications of this invention, it can be desirable to use nanoparticles having a variety of different diameters. Thus, one can manufacture a nanoparticle structure starting with particles having diameters of from about 20 nm to about 10,000 nm in the same reaction. Additionally, one can vary the shape of the particles to achieve improved heat transfer. By way of example, rod-shaped particles with a ratio of length to diameter of about 100:1 to 2:1, alternatively, from about 50:1 to about 5:1, and in other embodiments, about 20:1 to about 10:1.

Besides applications in optical spectroscopy (SERS, surface plasmon resonance spectroscopy, fluorescence, surface-enhanced infrared absorption spectroscopy and other spectroscopic techniques), fractal-like structures prepared from metal particles can be used as broad wavelength filters or antennae.

In addition, such systems can be used in a broad range of thermal-exchange methods and devices. For example, materials can be prepared from metal particles

arranged in a fractal-like structure, which will have superior thermal absorption - emission properties. These films can be used with heat-pipes for the removal of heat and integrated with materials for use in building construction, engine cooling systems, microchip cooling, and space technology. In addition, a clothing material could use this

5   invention for military purposes to obscure infrared detection (night vision).

B.   Manufacture of Nested Particle Associates

A nested particle associates can be made by selecting colloidal solutions of metal gold particles of uniform size, being 10 nm, 40 nm and 240 nm in diameter, respectively.

10   A plurality of 10 nm gold particles having a linker, such as DNA for example, can be attached thereto. A plurality of 40 nm particle is produced, each having a linker, such as DNA complementary to DNA linker of the 10 nm particle. Mixtures of the linker-derivatized 10 and 40 nm particles are placed in solution and interact with each other. The DNA linkers bind to each other to form a first-order nested structure.

15   A second-order nested particle structure comprising a plurality of first-order particle structures surrounding a larger particle larger than either of the first two particles, such as 240 nm. Heating the mixture of first-order particles or second-order particles to a temperature less than about 100° C and then cooling the mixture can result in better ordering of the nested particles.

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C.   Manufacture of Surfaces Having Non-Random Particle Structures

To manufacture heat pipes of this invention, one can deposit either chemically derived or nested particle structures onto a thermally conductive surface. The particle structures can desirably be affixed to the surface using a conductive polymer. When the

25   so-coated surface is exposed to electromagnetic radiation, some of the incident radiation is absorbed by the particle structures and can be transmitted to an interior portion of the heat pipe. The heat inside the pipe can then be transmitted along the heat pipe to another location, where, for example a cooler environment exists, thereby permitting the heat in the heat pipe to leave the heat pipe into the surrounding environment.

30   In certain other embodiments of this invention, nested or other particle structures in suspension can be used. These structures can be first attached and then a polymer can

be used to strengthen the attachment of the particle structures to increase the durability of the surface covering..

III. Thermally Conductive Polymers

Thermally and environmentally stable polymers such as polyaniline- or polypyrrole-based composites are capable of heat conduction (e.g., from EEONIX corporation). Other composite materials capable of attachment of metal particles to a metal surface are also well known (U.S. Patent 5,925,467, incorporated herein fully by reference).

Example 4
Heat Pipe Embodying Fractal Heat Absorber

Figure 3 depicts a portion **110** of an embodiment **300** of this invention in which a heat pipe **105**, similar to that shown in Figure 1, is partially covered with a particle structure. A heat absorber portion **110** comprises a heat pipe **105**, a wick element **135** having a volatile fluid **130**. A portion of volatilized fluid **140** is shown adjacent to wick **135**. Surrounding heat pipe **105**, a layer of heat conductive polymer **145** having metal particles **150** imbedded therein. Surrounding heat conductive polymer layer **145**, is a region **160** comprising nanoparticle structures.

Figure 4 depicts an embodiment of this invention **400** having a particle structure at a heat absorbing end **110** of a heat pipe. Heat pipe **105** has wick elements **135** having a volatile fluid **130** contained therein. The exterior of the heat pipe is shown having a layer of heat conductive polymer **145**, which has a region of particle structures **160** applied thereon. Heat (Qin) is absorbed by particle structure **160** at heat absorber portion **110** of the heat pipe and is transmitted to the heat pipe **105** by the polymer layer **145**. The heat vaporizes the volatile fluid **130** in wick region **135**, creating vaporized fluid **140**, which flows to the emissive end **120** of the heat pipe. At the emissive end **120** of the heat pipe, the vaporized fluid **140** condenses, releasing heat. The liquid flows into the wick structure **135** and is drawn back to heat absorber portion **110** of the heat pipe.

Figure 5 depicts an embodiment of this invention **500** having particle structures at both a heat absorbing end **110** and at an emissive end **120** of the heat pipe. The same

features described above in Figure 4 apply to Figure 5. Additionally, at emissive end 120 a layer of heat conductive polymer 146 surrounds heat pipe 105. A layer of particle structures 161 is applied to the exterior of heat conductive polymer layer 146. The types of heat conductive polymer 146 need not be the same as heat conductive polymer 145,

5   and the particle structure 161 need not be the same as particle structures 160. Heat transferred from the absorptive end 110 of the heat pipe 500 can be carried to the emissive end 120. At the emissive end 120, the volatilized fluid 140 can condense, releasing heat "Qout" which can flow through the heat conductive polymer layer 146, to the nanoparticle structure 161, that can thereby be radiated away.

10   In other embodiments of this invention, particle structures can be attached directly to the surface of a heat pipe without an intervening layer of heat conductive polymer. By way of example only, photolithographic methods can be used to attach the particle structures. Such methods are described in U.S. Patent Application, titled "Amplification of Analyte Detection by Substrates having Particle Structures with Receptors", Inventors:

15   David I. Kreimer, Ph.D., Oleg A. Yevin, Ph.D., Thomas H. Nufert, filed March 23, 2001, incorporated herein fully by reference. Furthermore, laser ablation can be used to manufacture particle structures directly on a heat pipe. These methods are described in the above cited U.S. Patent Application.

       In certain embodiments, it can be desirable to provide two ends of a heat pipe

20   with fractal aggregates having differing wavelength absorption/emission characteristics. For example, if an interior space has undesirably large heat production, it can be desirable to provide an end of a heat pipe with particle structures or fractal aggregates having high absorptivity at longer wavelengths characteristics of the heat within the space. At the other end, it can be desirable to provide particle structures or fractal aggregates having

25   emissive properties suited to radiative heat loss under those conditions.

IV   Window Materials Having Fractal Aggregates

       In certain embodiments of this invention, window materials, including for example, glasses, quartz, plastics and other materials known in the art can   be

30   temporarily treated with particle structures, including fractal aggregates to provide and/or control radiation absorptivity. To manufacture such windows, a solution containing

particle structures as described above can be applied to a surface by spray or by other methods for liquid dispensation. It can be desirable to provide the solution with an adhesive to promote adherence of the particle structures to the surface. As described herein, numerous adhesives can be used to attach particle structures.

In other embodiments, particle structures can be chemically attached to the surface of the window material using a chemical linker, such as silanes, including SiH_4. Window materials can be prepared by pre-cleaning, and then a chemical linker such as silane can be applied and then particle structures can be added to the pre-treated surface.

In certain embodiments in which the transmission of visible wavelengths is desired, but the transmittance of infrared wavelengths is not, the layer containing the particle structures should be relatively thin. Depending on the components of the solution, it is within the scope of the art to determine how thick a layer should be to provide a desired optical transmission.

Example 5
Preparation of Fractal Silver Aggregates using Citrate

Upon dissolving 45 mg $AgNO_3$ (Sigma) in 250 ml triple distilled, deionized water, the solution was brought to boiling under stirring with magnetic stirrer. A solution of 1 % sodium citrate (5 ml) was added drop-by-drop to the boiling solution under vigorous stirring. The solution was kept boiling for 60 min, while a tap water cooled condenser prevented loss of water due to evaporation. The resulting solution of colloidal silver was kept in a dark glass bottle at 5° – 8° C for a period of up to at least several months.

Fractal aggregates of silver particles were produced by mixing 250 microliters of the colloidal silver solution with 1750 microliters of a water solution of NaCl (Sigma, ultra pure) to bring the final concentration of NaCl to 60 mM. The aggregates formed within several minutes and the solution was fairly stable for at least about half an hour. Fractal aggregates were applied to a metal coated substrate, and the fractal aggregates sedimented and adhered to the metal surface, forming a fractal associate-derivatized metal surface.

Example 6
Preparation of Fractal Silver Aggregates using NaBH$_4$

A solution of 5 mM AgNO$_3$ (Sigma) in triple distilled, deionized water (50 ml)

5   was added drop-by-drop to 150 ml ice-cold, 2 mM solution NaBH$_4$ (Sigma),under vigorous stirring with magnetic stirrer. Right after that, a solution of 1 % poly(vinyl alcohol) (25 ml) was added. The mixture was then is boiled for 60 min, while a tap water-cooled condenser prevented loss of water due to evaporation. The solution of colloidal silver was kept in a dark glass bottle at 5° – 8° C for a period of up to at least

10   several weeks.

Fractal aggregates of silver particles were produced by mixing 250 microliters of the colloidal silver solution with 1750 microliters of a water solution of NaCl (Sigma, ultra pure) to bring the final concentration of NaCl to about 100 mM. The aggregates formed within several minutes and the solution was fairly stable for at least up to about

15   half an hour. Fractal aggregates were applied to a metal coated substrate, and the fractal aggregates sedimented and adhered to the metal surface, forming a fractal associate-derivatized metal surface.

Example 7
Preparation of Fractal Gold Aggregates Using Citrate

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Upon dissolving 120 mg HAuCl$_4$ (Sigma) in 250 ml triple distilled, deionized water, the solution was brought to boiling under stirring with magnetic stirrer. A solution of 1 % sodium citrate (5 ml) was added drop-by-drop to the boiling solution under

25   vigorous stirring. The solution was kept boiling for 60 min, while a tap water-cooled condenser prevented loss of water due to evaporation. The solution of colloidal gold was kept in a dark glass bottle at a temperature of about 5° – 8° C for up to at least about several weeks.

Fractal aggregates of gold particles were produced by mixing 250 microliters of

30   the colloidal gold solution with 1750 microliters of a water solution of NaCl (Sigma, ultra pure) to bring the final concentration of NaCl to ca. 100 mM. The aggregates formed within several minutes and the solution is stable for at least about half an hour. Fractal

aggregates were applied to a metal coated substrate, and the fractal aggregates sedimented and adhered to the metal surface, forming a fractal associate-derivatized metal surface.

Example 8
Window Having Fractal Absorber

For example, Figure 6a depicts perspective view of an embodiment **600** of this invention in which a piece of glass **604** has a top surface **608**. On the surface **608**, a layer **612** is provided, having an adhesive **616** and fractal aggregates **620**. Figure 6b depicts a cross-sectional view of an embodiment **600** as shown in Figure 6a having a piece of glass **604** having a top surface **608**. ON surface **608**, a layer **612** is provided, having adhesive layer **616** and fractal aggregates **620**.

Figure 7 depicts results of experiments to determine the optical absorbance properties of glass having silver fractal associates thereon. A pre-cleaned glass slide was obtained commercially and was further cleaned using isopropanol. Another slide so treated was exposed to a solution containing saturated diphenylsilanediol in isopropanol for 1 hour at room temperature (about 22° C). During the exposure, the surface was gently rubbed with a pledget of paper. The unbound silane was removed by rinsing with an excess of isopropanol. Another slide treated with silane was exposed to a colloidal solution containing silver fractal particles, prepared as in Example 5 above. After exposure to the colloid for 50 minutes, the excess particles were removed by rinsing with an excess of isopropanol followed by rinsing with an excess of water. Optical absorbance was measured over a range of wavelengths from about 250 nm to about 850 nm in a spectrophotometer.

In Figure 7, the wavelength of incident light in nm is presented on the horizontal axis, and the absorbance in optical density (OD) units of treated and untreated glass is presented on the vertical axis. Untreated glass (bottom trace) has the lowest optical density at all wavelengths tested. For most wavelengths above about 250 nm, the optical density is about constant, with a slight decrease from about 250 nm to about 850 nm. Glass having a coating of silane exhibits somewhat greater optical density than untreated glass, but the difference in optical density between untreated and silane-treated glass is

slight.   However, glass having silane and silver fractal aggregates together exhibits greater absorbance at most wavelengths tested.

A range of increased transmittance (decreased absorbance) was observed in a range of wavelengths of about 280 nm to about 350 nm.   Within this range, the transmittance approached those of either untreated glass or silane-treated glass.

Figures 8a - 8b depict yet other embodiments of this invention in which particles structures are embedded within the window material. Figure 8a depicts a "sandwich" embodiment **800** in which a first piece of window material **804** has a layer **808** having particle structures **812** therein. A second piece of window material **816** is provided on layer **808**, thereby completing the sandwich. It can be appreciated that layers **804** and **812** need not be the same thickness or even material for the improved radiation absorptive properties to be useful.

Figure 8b depicts an alternative embodiment **802** in which a single piece of window material **820** has particle structures **824** distributed throughout the piece of material. One can readily appreciate that such "distributed" embodiments can comprise a variety of thicknesses, and in yet other embodiments, multiple layers of "distributed" embodiments can be combined to provide multilayered structures having desirable radiation absorptive properties.

One can also appreciated that variations of the above embodiments are within the scope of this invention. For example, one can provide mixtures of particle structures, each component of the mixture having different absorptive properties. By using such mixtures, broader ranges of radiation absorptivity can be provided.

In certain embodiments, the particle structures can be adhered to the surface of a transparent material using a soluble matrix, such as a glue. If the matrix is soluble in common solvents, such as water, alcohol, or an ammoniacal cleaning solution, then the particle structures can be removed when the absorptive properties of the particle structures are no longer desired. One example of such a soluble adhesive is starch. Alternatively, the particle structures can be adhered to the surface more permanently, using matrices that are not easily dissolved. In yet other embodiments, the particle structures can be adhered to the surface using one or more insoluble polymers, as described herein above.

Additionally, in certain embodiments, particle structures can be provided in a matrix in the form of a tape having a stick backing that can be applied to a surface of a piece of window material. Such embodiments are herein termed "window coverings." Such window coverings can be provided in bulk, for example in rolls either having or not having a release liner. As used herein, the term "release liner" means a piece of material that adheres weakly to the adhesive of the tape. Thus, the tape having particle structures can be protected from undesired consequences of the adhesive adhering to the wrong side of the particle-containing matrix. Such release liners are known in the art.

Figure 9a depicts an embodiment of this invention **900** in which a sheet of polymer material **904** has particle structures **908**, and a layer of adhesive backing **912**. A release liner **916** is applied to the adhesive backing **912**.

In yet other embodiments, window materials of this invention comprise particle structures and other absorptive materials, such as dyes. Such dyes include tinting materials commonly known in the art of window manufacture, and can confer color and/or polarizing properties to the window material. Figure 9b depicts an alternative embodiment **902** similar to that shown in Figure 9a and having an additional layer **920** having polarizing properties.

The embodiments described and depicted are for illustration only, and are not intended to limit the scope of the invention. Numerous variations of the surfaces having particle structures are within the scope of persons of skill in the arts, and all of those variations are included within the scope of this invention.

V.   Use of Particle Structures in Analytical Instruments

In certain embodiments of this invention, window materials can be prepared with particle structures to improve the characteristics of sample holders for analytical instrumentation. By way of example, in Raman spectroscopy, a substrate having an analyte of interest can be exposed to incident electromagnetic radiation, and Raman scattering can produce a Raman spectral feature. in certain circumstances, the wavelength of a Raman spectral feature can be masked by electromagnetic radiation arising from other sources than the analyte. This "parasite light" can be a problem,

especially for analytes having weak Raman spectral features. Other types of optical detection systems that suffer from parasite light include phase optics, fluorescence optics, and any other type of detection/analysis system that uses electromagnetic radiation.

To overcome the problem of parasite light, devices of this invention can be made that incorporate the particle structures described herein. By selectively filtering out parasite light, the identification of signals can be improved.

INDUSTRIAL APPLICABILITY

The particle structures of this invention are useful for the manufacture of improved absorptive and emissive materials used for heat pipes and windows. Selection of wavelengths absorbed and/or transmitted through window materials can be based upon the environmental conditions on the two sides of the widow. Improved heat absorption can keep the interior of structures within desired temperature ranges. Heat pipes having improved heat absorptive and transmissive surfaces can maintain proper operating conditions for a variety of equipment and spaces, including structures. Application of the particle structures of this invention can improve the efficiency of heat pipes.